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(54) SYNTHETIC FIBRES

(71) We, POLYSAR LIMITED, a company organised under the laws of Canada, of Sarnia, Ontario, Canada, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process for the production of a synthetic fibre from the melt. In particular, the fibre of this process is an acrylonitrile-containing material.

Fibres made from polymers of acrylonitrile have long been known in the art. Such polymers usually contain at least 75% by weight of acrylonitrile, frequently at least 85%, and most usually 90—99%, of acrylonitrile. Typical monomers copolymerized with the acrylonitrile include vinyl pyridine, styrene, vinyl chloride and vinyl acetate, each monomer when copolymerized with the acrylonitrile causing a change in one or more properties of the fibre, especially dye-ability. Such acrylonitrile polymers are formed into fibres by spinning from a solution of the polymer in a suitable solvent or from a suspension of the polymer in a suspending agent. They cannot be spun into fibres from the melt. In order to achieve reasonable strength or tenacity in the fibre it usually has to be after-stretched, which leads to the development of orientation in the polymer molecules in the fibre, the stretching being at an elevated temperature and by at least a 100—200% increase in length, followed by cooling of the fibre.

It has now been discovered that a synthetic fibre may be produced by spinning from the melt of a polymer of acrylonitrile, styrene and isobutylene. The fibre so produced has a high tenacity without the need for being after-stretched.

It is an object of this invention to provide a process for the production of a synthetic fibre by spinning from the melt of a polymer of acrylonitrile, styrene and isobutylene.

The polymers used to produce the fibres are polymers which preferably contain from 65 to 75 weight % of acrylonitrile, from 13 to 18 weight % of styrene and from 13 to 18 weight per cent of isobutylene. More preferably the polymer contains from 66 to 71 weight % of acrylonitrile, from 14 to 17 weight % of styrene and from 14 to 17 weight % of isobutylene. Such polymers are advantageously prepared by a free radical aqueous emulsion polymerization process and recovered from the aqueous phase by conventional coagulation techniques. The recovered water-wet polymer is dried, for example, in a forced-air dryer at a temperature of at least about 180°F. For convenience in subsequent use of the polymer, the product from the dryer may be further treated by passage through a vacuum extruder in order to reduce residual water to a minimum and the extruded product may then be pelletized.

Such a polymer may be used to prepare fibres. Because polymers containing high levels of acrylonitrile tend to absorb moisture on storage, it is desirable to vacuum dry the pelletized polymer immediately prior to forming into fibres. The so dried polymer is then supplied to an extruder for formation into filaments. The extruder is operated at a high temperature to melt the polymer and to cause good flow of the molten polymer. The product from the extruder exits through a spinning pack filter system before passing to a spinneret which contains a number of small holes of fixed dimension. Such spinnerets are well known in the industry. Temperatures of about 350°F to about 500°F are suitable for the operation of the extruder, spinning pack filter system and spinneret. Preferably, the temperature at which the extruder is operated is from 350°F to 425°F. A preferred temperature range for the operation of the spinning pack filter system is from 400° to 500°F; pressure in the spinning pack filter system will usually be from 1,000 to 3,500 psi and preferably from 2,000 to 3,000 psi. The

filaments flowing from this spinneret are cooled, suitably by one of cool gas or liquid and most suitably by cool air. The rate of cooling is usually controlled at a uniform rate. The cooled filament is then wound onto a suitable bobbin. This windup process may be used to cause draw-down of the filament or it may be wound up without imparting any stretch to the filament. The windup may be at a constant speed or at a constant tension. When the filament is subjected to draw-down the useful range of draw-down ratios is from 10:1 to about 100:1, with a preferred range being from about 20:1 to 50:1. The denier of the filament may range from about 5 up to about 100 or more, depending upon the use to which the final fibre is going to be put. The wound up filament or fibre may then be handled in a variety of manners depending on the use to be made of the fibre. The fibre may be subjected to after-stretching, either hot or cold, to induce a greater degree of orientation within the fibre and thereby impart higher strength, it may be cut into short lengths or it may be used directly in a weaving process, either alone or blended with other fibres. The fibre of this invention has sufficient strength that it may be used without after-stretching either alone or blended with another fibre, for instance polypropylene. The polymer may also be alloyed with another fibre-forming material such as polypropylene, and the alloy then formed into fibres. The fibre of this invention has good elongation properties as shown by good knot tensile strength. The fibre has also been found to have good temperature stability as determined by simple ironability tests. The commercial advantages of spinning a fibre directly from the melt are well known in the industry.

The following example serves to illustrate the invention and not to limit the scope thereof.

EXAMPLE 1

Polymer Preparation

A polymer suitable for forming into fibres was prepared by an emulsion polymerization process. To a reactor equipped with inlet means, agitating means and temperature regulating means was added 200 parts by weight of water, 2 parts by weight of the di-sodium salt of a straight chain ethoxylated alcohol half ester of sulfosuccinic acid, 2 parts by weight of sodium mono alkyl-phenoxy benzene disulphonate, 0.2 parts by weight of sodium bisulphite and 15 parts by weight of isobutylene. The temperature of the contents of the reactor was raised to 122°F and the agitation means was put into operation. In a separate

container was prepared a mixture of 70 parts by weight of acrylonitrile, 15 parts by weight of styrene and 0.5 parts by weight of tertiary dodecyl mercaptan. Also in a separate vessel was prepared a 0.5 weight % solution of potassium persulphate in water. Sufficient potassium persulphate solution was added to the reactor to correspond to 0.5 parts by weight of potassium persulphate. Essentially simultaneously the addition was started of the acrylonitrile/styrene/mercaptan mixture, the rate of addition being uniform and such that the addition was complete after a total of five hours. At four hours from the start of the addition of the acrylonitrile/styrene/mercaptan mixture, an increment of potassium persulphate solution, corresponding to 0.25 parts by weight of potassium persulphate was added to the reactor. The polymerization was stopped at seven hours after the start of the addition of the polymerizable monomers, the conversion of total monomers to polymer being 92%, an aqueous slurry of alkylated aryl phosphite was added to the latex in an amount equivalent to 1 part by weight per 100 parts by weight of polymer. The polymer was recovered by adding the latex to a 1% solution of calcium chloride at a temperature of 190°F and separating off the coagulated polymer which was then water washed and dried in a forced air dryer at a temperature of 180 to 190°F. The so dried polymer was then further dried by being put through a vacuum extruder and the extruded product was pelletized. The polymer was found to contain, by weight, 69% of acrylonitrile, 16% of styrene and 15% of isobutylene.

Fibre Preparation

The pelletized polymer was dried in a vacuum oven for 1 hour at 80°C and then fed to an extruder. The experimental fibre spinning system contained an extruder having a diameter of 1 inch and an L:D ratio of 25:1 with a three-zone temperature control system on the barrel. The output from the extruder was fed through a valve adapter to a spinning pack containing a 100 mesh screen and into a spinneret having 104 holes of 0.02 inch diameter. The filament from the spinneret was cooled by a stream of air and passed to a windup bobbin. The extruder was operated at a screw speed of 18 rpm and at a temperature of 360°F on all three barrel sections and the spinning pack was maintained at 485°F, the pressure in the spinning pack being 2,700 psi. The cooling air was maintained at a pressure of 10 psi and the windup bobbin was run at a speed such that the rate of windup was 48 ft. per minute. Draw-down of the filament from the spinneret was 40 to 1.

Fibre Properties

The fibre so produced had an average denier of 59 and an average diameter of 126 microns. The average tensile strength of the straight fibre was 1.9 grams per denier and the elongation to break was 27%. Knotted fibre had an average tensile strength of 1.5 grams per denier and an elongation of 16%, showing about 80% retention of the strength at the knot.

The fibres produced in this example were found to show little evidence of sticking to an iron when subjected to conventional ironing at medium temperature settings.

This example shows that a fibre having good strength properties, without having undergone after-stretching, is readily produced from the polymer of this invention.

The polymer may be alloyed with, for example, polypropylene in the extruder and a fibre produced from the alloy. Also the pure fibre may be blended with polypropylene fibres for other applications.

WHAT WE CLAIM IS:—

1. A process of forming a fibre which comprises spinning from the melt a polymer of acrylonitrile, styrene and isobutylene.

2. The process of claim 1 wherein said polymer contains from 65 to 75 weight % of acrylonitrile, from 13 to 18 weight % of styrene and from 13 to 18 weight % of isobutylene.

3. The process of claim 2 wherein said polymer contains by weight 66 to 71% of acrylonitrile, 14 to 17% of styrene and 14 to 17% of isobutylene.

4. The process of any preceding claim wherein said polymer is fed through an extruder to a spinning pack filter system to a spinneret.

5. The process of claim 4 wherein said extruder, spinning pack filter system and spinneret are operated at temperatures of from 350°F to 500°F.

6. The process of claim 5 wherein said extruder is operated at a temperature of from 350°F to 425°F and the spinning pack filter system at a temperature of 400°F to 500°F at a pressure of 1,000 to 3,500 psi.

7. The process of claim 6 wherein said spinning pack filter system is operated at a pressure of 2,000 to 3,000 psi.

8. The process of any one of claims 4 to 7 wherein the product from the spinneret is subjected to draw-down within the range of from 10 to 1 to 100 to 1.

9. The process of any preceding claim wherein the melt which is spun is a blend of the polymer of acrylonitrile, styrene and isobutylene with another fibre-forming polymer.

10. The process of claim 1 substantially as hereinbefore described with reference to the Example.

11. Fibres produced by the process of any preceding claims.

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